| Ref<br># | Hits | Search Query  | DBs                       | Default<br>Operator | Plurals | Time Stamp       |
|----------|------|---|---------------------------|---------------------|---------|------------------|
| L1       | 902  | 210/670   | USPAT;<br>EPO;<br>DERWENT | AND                 | ON      | 2005/09/30 15:22 |
| L2       | 253  | L1 and membrane   | USPAT;<br>EPO;<br>DERWENT | AND                 | ON      | 2005/09/30 17:01 |
| 13       | 12   | l1 and nanofiltration   | USPAT;<br>EPO;<br>DERWENT | AND                 | ON      | 2005/09/30 17:14 |
| L4       | 26   | ("3996131" "4329225" "4648976"<br> "4806244" "5182023" "5364534"<br> "5464530" "5595666" "5639377"<br> "5728302" "6020210" "6027649"<br> "6120688").PN. | USPAT;<br>EPO;<br>DERWENT | AND                 | ON      | 2005/09/30 17:30 |
| L5       | 15   | ("4207397" "4804465" "5254257"<br> "5932099" "6372143" "6464881"<br> "6783681").PN.   | USPAT;<br>EPO;<br>DERWENT | AND                 | ON      | 2005/09/30 17:38 |
| L6       | 4    | ("4154675" "6776913").PN.   | USPAT;<br>EPO;<br>DERWENT | AND                 | ON      | 2005/09/30 17:40 |
| L7       | 2    | ("6669849").PN.   | USPAT;<br>EPO;<br>DERWENT | AND                 | ON      | 2005/09/30 17:50 |
| L8       | 3    | "5772891"   | USPAT;<br>EPO;<br>DERWENT | AND                 | ON      | 2005/09/30 17:51 |
| L9       | 2    | ("5772891").PN.   | USPAT;<br>EPO;<br>DERWENT | OR                  | OFF     | 2005/09/30 17:51 |

US-PAT-NO:

5792441

DOCUMENT-IDENTIFIER: US 5792441 A

TITLE:

Fixed-resin bed technologies for the treatment of the

chlorine dioxide generator effluent and feeds stream

DATE-ISSUED:

August 11, 1998

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APPL-NO:

08/729148

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INT-CL:

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US-CL-ISSUED:

423/531, 423/522, 423/551, 423/DIG.14, 210/660, 210/670

US-CL-CURRENT: 423/531, 210/660, 210/670, 423/522, 423/551, 423/DIG.14

FIELD-OF-SEARCH: 423/551; 423/531; 423/522; 423/DIG.14; 210/660; 210/670

**REF-CITED:** 

U.S. PATENT DOCUMENTS

PAT-NO

ISSUE-DATE

PATENTEE-NAME

**US-CL** 

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**ART-UNIT:** 

113

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# **ABSTRACT:**

A process employing a fixed-resin bed Acid Retardation Unit (ARU) to separate spent acid from chlorine dioxide generators into a de-acidified sodium sulfate component and a purified sulfuric acid component. The de-acidified sodium sulfate produced is returned to the chemical recovery cycle of the kraft mill in place of acidic chlorine dioxide generator effluent thereby avoiding the destruction of alkalinity in pulp mill liquors. In addition, sulfuric acid is purified providing an opportunity for reuse in various mill applications, and/or concentrated and recycled to the generator. Alternatively, to avoid high evaporation costs, the purified acid can be used to regenerate a cation-exchange unit (CEU) used in the conversion of sodium chlorate to a sodium chlorate/chloric acid mixture which is fed to the generator in place of sodium chlorate and sulfuric acid. Using this approach, the sulfuric acid requirement of chlorine dioxide generators and, in turn, the amount of acidic generator effluent can be reduced. The process is applicable to both atmospheric and subatmospheric chlorine dioxide generators. In the latter case, the concentration of the purified sulfuric acid from the ARU or any other acid separation system can be increased by using a portion of it to dissolve the sodium sesquisulfate by-product from such generators.

20 Claims, 10 Drawing figures

Exemplary Claim Number: 1

Number of Drawing Sheets: 10

----- KWIC -----

Brief Summary Text - BSTX (30):

In none of the aforementioned systems, however, is suggestion made for the

application of suitable fixed-resin bed ion-exchange technology to the treatment of the spent acid from atmospheric or subatmospheric chlorine dioxide generators in such a way as to avoid the problems of the prior art; in particular no mention is made of using a fixed-resin bed acid retardation unit (ARU) to produce a de-acidified sodium sulfate solution that can be returned to the recovery cycle without destroying alkaline components in pulp mill liquors and a purified sulfuric acid stream that can be used in other mill applications including the regeneration of a fixed-resin bed cation-exchange unit (CEU) for the conversion of sodium chlorate to a sodium chlorate/chloric acid mixture prior to feeding it into the generator. Eventhough, Twardowski (U.S. Pat. No. 4,678,655, Jul. 7, 1987) describes a diffusion dialysis process for the de-acidification of spent acid from atmospheric chlorine dioxide generators, no suggestion is made for using such a system for subatmospheric generators. Furthermore, in none of the aforementioned systems dealing with subatmospheric generators is suggestion made of using the purified acid from a suitable fixed-resin bed ARU or any other acid separation process (e.g. diffusion dialysis, electrodialysis or nanofiltration) to dissolve the sodium sesquisulfate by-product from such generators in a way that would increase the concentration of the recovered acid to sufficiently high levels to enable its return to the chlorine dioxide generator.

# Brief Summary Text - BSTX (57):

(iii) the treated solution is introduced into a fixed-resin bed ARU or any other acid separation system (e.g. diffusion dialysis, electrodialysis, nanofiltration) thus producing a de-acidified sodium sulfate solution which is directed to the recovery cycle for sodium and sulphur make-up and purified sulfuric acid,

# Brief Summary Text - BSTX (94):

In this case, a slurry of sodium acid sulfate salt is produced as a result of reaction (1). This slurry is withdrawn and passed to a solid/liquid separation device such as a rotary drum vacuum filter. The acid filtrate is returned to the generator while the cake recovered from this filter is conveyed to a dissolving tank equipped with a mixer and heater to facilitate dissolution of the salt in the solution contained therein. Residual chlorate, chlorine dioxide and chlorine values are destroyed using a suitable reducing agent as described above in the case of atmospheric generators. The treated solution is then fed to a fixed resin bed ARU or any other acid separation system (e.g. diffusion dialysis, electrodialysis or nanofiltration). The de-acidified sodium sulfate solution is recycled to the kraft recovery cycle, (either the black or green liquor) for sodium and sulphur make-up. A portion of the purified acid withdrawn from the fixed-resin bed ARU or any other acid

separation system is recycled to the dissolving tank and used for the dissolution of sodium sesquisulfate. A second portion of the purified acid product is utilized for rinsing generator media from the filter cake on the vacuum filter and the remainder is recycled to the chlorine dioxide generator, at a sufficiently high concentration to minimize evaporation costs.

# Detailed Description Text - DETX (9):

FIGS. 8, 9 and 10 specifically apply to subatmospheric chlorine dioxide generators which produce solid sodium sesquisulfate as a by-product of the chlorine dioxide process. In this case, a fixed-resin bed ARU or any other acid separation system (e.g. diffusion dialysis, electrodialysis, nanofiltration) can be employed with this invention. For simplicity, discussion will be largely restricted to the fixed-resin bed ARU.

# Detailed Description Text - DETX (10):

FIG. 8 shows the solubility of sodium sulfate at different sulfuric acid concentrations at a temperature of 46.degree. C. Line AC represents the ratio of sodium sulfate to sulfuric acid present in sodium sesquisulfate, which corresponds to a sulfuric acid content of approximately 18% of the total. The point B at which line AC crosses the solubility line represents the maximum solubility of sodium sesquisulfate that can be achieved by dissolution in water. This will produce a solution containing a sulfuric acid concentration of about 7% and a sodium sulfate concentration of about 32%. If this solution were fed to a fixed-resin bed ARU or any other acid separation system (e.g. diffusion dialysis, electrodialysis, nanofiltration), the sulfuric acid would be removed from the feed solution, thus producing a de-acidified sodium sulfate solution. The maximum concentration of sulfuric acid that can be withdrawn from the fixed resin bed ARU or most other acid separation systems would be that of the feed i.e. 7%. In practice, the actual acid concentration would be somewhat less, typically 5-6%, due to loss of acid values to the de-acidified salt by-product solution.

# Detailed Description Text - DETX (12):

FIG. 10 is another embodiment of the invention which illustrates how sodium sesquisulfate from subatmospheric chlorine dioxide generators can be separated into a de-acidified sodium sulfate stream that can be returned to the kraft recovery cycle and a purified sulfuric acid stream of sufficiently high concentration to be returned to the chlorine dioxide generator. In such a generator, 100, a slurry of sodium acid sulfate salt, 801 is produced as a result of reaction 1. This slurry is withdrawn and passed to a solid/liquid separation device, 802 such as a rotary drum vacuum filter. The acid filtrate, 803, is returned to the generator. The cake recovered from this filter is

conveyed via connecting means, 804 to a dissolving tank, 805 equipped with a mixer and heater (not shown) to facilitate dissolution of the salt in the solution contained therein. In this case, the destruction of residual chlorate, chlorine dioxide and chlorine values can be carried out in the dissolving tank. The treated solution is fed to a fixed-resin bed ARU or any other acid separation system (e.g. diffusion dialysis, electrodialysis, nanofiltration). The sulfuric acid is thus removed from the feed solution vielding a de-acidified sodium sulfate solution, 211, which can be recycled to the kraft recovery cycle, 300 (either the black or green liquor) for sodium and sulphur make-up. Because this solution is de-acidified, the caustic make-up, 301 to the recovery cycle (white liquor) is expected to be reduced significantly. If a fixed-resin bed ARU is employed, fresh water, 212 is used to wash off the ARU resin bed a purified sulfuric acid product, 213; alternatively, such an acid product can be withdrawn from any other acid separation system. A portion of the purified acid product, 213, is recycled to the dissolving tank, 805 via connecting means, 806. A second portion of the purified acid product, 807 is utilized for rinsing generator media from the filter cake on the vacuum filter, 802 and the remainder, 808 is recycled to the chlorine dioxide generator, 100.

Detailed Description Text - DETX (68):

This example provides a way to utilize a fixed-resin bed ARU or other acid separation systems (e.g. diffusion dialysis, electrodialysis, nanofiltration) to recover sulfuric acid from sodium acid sulfate salts produced from chlorine dioxide generators, while increasing the concentration of the recovered sulfuric acid.

Issued US Cross Reference Classification - CIXR (5): 210/670

Field of Search Class/SubClass - FSCS (6): 210/670

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L6: Entry 2 of 5 File: USPT Dec 21, 1999

DOCUMENT-IDENTIFIER: US 6004464 A

\*\* See image for Certificate of Correction \*\*

TITLE: Spent brine reclamation

#### Abstract Text (1):

A method of reclaiming spent aqueous brine solutions used in the regeneration of water-softening <u>resins</u>. An aqueous solution chloride or other brine is acidified with HCl to a pH of between about 0.5 and 6 and a soluble sulfate salt, preferably Na.sub.2 SO.sub.4 is added, together with a precipitation inhibitor of the type polyacrylamide. Following such treatment, the spent brine is pumped at high pressure axially through a spirally-wound <u>nanofiltration</u>-type membrane device which is effective to remove at least about 90% of the divalent hardness while allowing passage therethrough of at least about 90% of the monovalent cations. As a result of such treatment, about 90 to 95% of the volume of spent brine can be efficiently and effectively reclaimed in a form in which it is suitable for use again in regenerating water-softening resins or the like.

#### Brief Summary Text (1):

The invention relates generally to reclaiming spent aqueous brine solution by removing divalent cations by the use of a semipermeable membrane, and more particularly to the reclamation of spent brine solutions that result from the regeneration of water-softening resins or the like so as to facilitate the reuse of a major portion of such spent brine and significantly reduce waste discharges from such a water-softening resin regeneration operation.

#### Brief Summary Text (3):

Water softeners and the like use ion exchange <u>resins</u>, such as zeolite <u>resins</u>, to remove polyvalent ions from aqueous streams by exchanging them with monovalent cations such as sodium. When there are few sodium ions left on such <u>resins</u>, it becomes necessary to regenerate the ion exchange <u>resins</u> by treatment with an aqueous brine containing sodium chloride or the like, e.g. potassium chloride. As a part of this regeneration, the polyvalent cations are solubilized and replaced on the <u>resin</u> with sodium ions. The resultant aqueous solution contains some sodium chloride plus the solubilized polyvalent ions and is generally referred to as "spent brine". Although heretofore it has usually been acceptable to simply discharge such spent brine into a municipal sewer system, environmental restraints are being imposed throughout many parts of the United States, and the need to conserve water has also dictated that efforts be made to reclaim such spent brine.

# Brief Summary Text (4):

In May of 1992, Larry Lien of Membrane Development Specialists, Inc. was asked to speak at the semiannual meeting of the Pacific Water Quality Association in Santa Barbara, Calif. At that meeting, the concept of reclaiming spent brine used to regenerate water-softening resins was recommended, and it was suggested a semipermeable membrane separation treatment, specifically one using spirally-wound elements or cartridges made of the DS-5 nanofiltration membrane being marketed by Desalination Systems, Inc., might be advantageously employed. The adoption of such spent brine reclamation systems was advocated so as to reduce the cost of sodium chloride and reduce sewer charges, as well as because it would be an environmentally sound practice.

## Brief Summary Text (6):

In January of 1993, a United States patent application was filed, which issued later that year as U.S. Pat. No. 5,254,257. It is directed specifically to the concept of purifying spent brine from the regeneration of ion exchange <u>resins</u>. The patent discloses the use of DS-5 nanofiltration membrane cartridges for spent brine reclamation by first acidifying the spent brine with hydrochloric acid to a pH of about 1.5 to 2.4 and subsequently treating the purified or reclaimed brine with sodium hydroxide to raise the pH.

#### Brief Summary Text (10):

It has now been found that spent aqueous brine solutions from the regeneration of water-softening resins or the like can be efficiently and effectively reclaimed by treating an acidified solution with a soluble sulfate salt and a precipitation inhibitor prior to applying the treated solution to a nanofiltration membrane which is designed to selectively reject divalent and other polyvalent cations. Preferably, the spent brine is acidified to a pH of between about 0.5 and about 6 and is treated with a soluble sulfate, such as sodium, potassium, lithium or ammonium sulfate or hydrogen sulfate (sometimes termed bisulfate), in an amount at least about equal to one-half the total weight of the calcium and magnesium ions contained therein. The precipitation inhibitor is preferably of the polyacrylamide type, such as that which is sold as Cyanamer P-70. By employment of a spirallywound nanofiltration cartridge which has feed-channel-providing spacer material having a thickness of at least about 1 mm, the treated brine can be efficiently pumped to a pressure between about 600 and about 1,000 psi and then applied to the cartridge, at which pressure it will have an axial velocity of at least about 10-15 cm per second; as a result, it is found to be efficient and economical to recover about 90 to 95 volume % of the spent brine that is being treated having less than 10% of the original hardness, thus reducing the amount of waste to be discharged to a sewer or otherwise disposed of by as much as a factor of about 20. Moreover, brine containing TDS over 100,000 ppm and hardness of at least 17,000 ppm can be reclaimed as brine with hardness of not more than 1,000 ppm.

## <u>Drawing Description Text</u> (2):

FIG. 1 is a schematic view of a system which may be employed in a method embodying various features of the invention that is designed to reclaim spent brine obtained from the regeneration of water-softening <u>resins</u>.

#### Detailed Description Text (3):

Illustrated diagrammatically in FIG. 1 is a representative system for recovering spent brine that was used for the regeneration of water-softening resins or the like, so that such brine is now contaminated with relatively high amounts of hardness, primarily in the form of divalent calcium and magnesium cations and to a lesser extent barium cations. While the illustrated system is designed for batch operation, one having the ordinary skill in this art will realize that it can easily be adapted to continuous operation, if desired, where recovery of about 85% or higher of the feed volume is easily achieved.

# Detailed Description Text (4):

Shown is a brine feed tank 11 wherein an initial quantity of spent brine, for example 1,000 gallons, would be collected. The brine is acidified to a pH between about 0.5 and about 6, and preferably to a pH between about 2 and about 5. Preferably, hydrochloric acid is used for the acidification; however, other strong acids that may alternatively be used include sulfuric, nitric and phosphoric. The spent brine to be reclaimed is analyzed for hardness; typical spent brines from the regeneration of water-softening resins may contain total dissolved solids (TDS) in the range of about 100,000 to 150,000 ppm, of which hardness (measured as calcium carbonate) may measure from 10,000 to 25,000 ppm. Average values might be considered to be about 17,500 to 19,000 ppm of hardness measured as CaCO.sub.3. An appropriate precipitation inhibitor or antiscalant is also added to facilitate high

recovery. The preferred antiscalant is one of the polyacrylamide chemical family, one example of which is sold under the tradename Cyanamer P-70 by the American Cyanamid Company. Other antiscalants based upon polyacrylic acid or polymalic acid, as are well known, may be used. Preferably, such an antiscalant is used in an amount between about 5 and about 10 ppm.

## Detailed Description Text (11):

It has been found that, by treating the feed brine with an appropriate amount of a soluble sulfate, preferably sodium sulfate, and an antiscalant, it is possible to operate the illustrated system to efficiently process brine containing substantial amounts of hardness and recover 90 to 95% of the spent brine in a form which is considered suitable for reuse in the regeneration of water-softening <u>resins</u>.

#### Detailed Description Text (12):

As examples of the efficiency of such a system, two spirally-wound nanofiltration elements made with DS-5 semipermeable membrane, which are 40 inches in length and 4 inches in diameter, are employed in a series relationship as shown in FIG. 1. The spent brine to be treated has TDS of about 130,000 mg/l, including about 19,000 mg/l of hardness. The pH of the spent brine is lowered with HCl to about 5.1, and sodium sulfate is added to provide a sodium sulfate level of about 1,500 ppm. Cyanomer P-70 antiscalant is added to provide a level of about 7.5 ppm. The high pressure pump 25 is operated to apply a pressure of about 800 psi to the liquid stream entering the first of the spirally-wound nanofiltration cartridges. An average flux equal to about 9 gallons per square foot per day is achieved using these two spirally-wound nanofiltration cartridges. The entire batch of spent brine is processed until about 90% of the original amount has become clean brine product, so that the brine feed tank now contains only about 10% of the original volume. Examination of the clean brine shows that it has a hardness of only about 4,000 ppm; this indicates that about 91% of the original hardness is removed as a result of the processing. The resultant recovered purified brine in the tank 55 is considered to be fully suitable for use in regenerating spent water-softening resins.

# Detailed Description Text (14):

As a further example, a single 8-inch diameter, 40-inch long spirally-wound nanofiltration element of DS-5 membrane is used to purify a spent sodium chloride brine having hardness equal to about 17,500 ppm, measured as calcium carbonate. The pH is adjusted with HCl to about 0.1 molar equivalent, and based upon the measured hardness of 17,500 ppm, sodium sulfate is added in an amount equal to about 5,000 ppm. Cyanomer P-70 is added at a level of about 7.5 ppm. To process a batch of about 1,000 gallons of such treated spent brine, a high pressure pump is employed to feed the treated brine to the 8-inch diameter element at a pressure of about 800 psi. The feed spacer in the 8-inch element has a thickness of about 2.1 mm, and operation of the pump maintains a velocity of about 10-15 cm per sec in the axial flow path through the element. At this pressure, the operation of the 8-inch diameter element produces a flux of about 2,000 to 2,500 gallons per day, based upon 24-hour operation, and operation is continued until about 950 gallons is present in the product clean brine tank and only 50 gallons remain in the feed tank. Testing shows that the hardness removal of the system is excellent, and the clean brine has a hardness of about 4,000 ppm and is considered satisfactory for use in the regeneration of water-softening resins.

#### CLAIMS:

- 1. A method of reclaiming an aqueous brine solution used in the regeneration of water-softening resins, which method comprises the steps of:
- (a) treating an aqueous NaCl or KCl brine solution that contains significant amounts of divalent cations, by adding (i) a soluble sulfate salt, (ii) with an acid selected from the group consisting of hydrochloric, sulfuric, nitric and

phosphoric to lower the pH to about 0.5 and about 6, and (iii) a precipitation inhibitor selected from the group consisting of polyacrylic acid, polyacrylamide and polymalic acid;

- (b) applying said treated brine from step (a) to semipermeable membrane material which permits the passage therethrough of at least about 90% of monovalent ions in said solution while rejecting at least about 90% of all divalent cations in said aqueous solution; and
- (c) recovering the permeate from step (b) for use in regenerating water-softening resins.  $\bullet$
- 13. A method for reclaiming a batch of aqueous brine used in the regeneration of water-softening resins or the like, which method comprises the steps of:
- (a) treating a batch of aqueous NaCl or KCl brine containing hardness in the form of Ca.sup.++ and Mg.sup.++ cations in a tank by adding soluble sulfate, a strong acid, and a precipitation inhibitor,
- (b) withdrawing said treated brine from the tank and pumping same through a spirally-wound semipermeable membrane cartridge to create a permeate stream and a concentrated stream, which membrane permits the passage therethrough of at least about 90% of monovalent ions in said brine while rejecting at least about 90% of all divalent cations in said brine, so
- (c) recovering said permeate stream from the semipermeable membrane cartridge while returning at least a portion of said concentrate stream to said tank, and continuing said withdrawing of treated brine from said tank and pumping same through said cartridge until such time as at least about 90 volume % of said brine has been recovered as permeate containing less than 10% of Ca.sup.++ and Mg.sup.++ cations present in said batch of brine that was initially treated in step (a).
- 15. A method for reclaiming a batch of aqueous brine used in the regeneration of water-softening  $\underline{\text{resins}}$  or the like by removing hardness therefrom, which method comprises the steps of:
- (a) treating aqueous NaCl or KCl brine solution containing hardness in the form of Ca.sup.++ and Mg.sup.++ cations by adding soluble sulfate, a strong acid, and a precipitation inhibitor so that said solution has a pH between about 0.5 and about 6,
- (b) pumping said treated brine from said tank through a spirally-wound semipermeable membrane cartridge to create a permeate stream and a concentrated stream, which membrane permits the passage therethrough of at least about 90% of monovalent ions in said brine while rejecting at least about 90% of all divalent cations in said brine, so
- (c) recovering said permeate stream from the semipermeable membrane cartridge while recirculating at least a portion of said concentrate stream, and continuing said pumping of treated brine through said cartridge including said portions being recirculated so that recovery of at least about 80 volume % of said brine that is treated is recovered as permeate containing less than 10% of Ca.sup.++ and Mg.sup.++ cations present in said initially treated brine.

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L6: Entry 4 of 5 File: USPT Aug 3, 1999

DOCUMENT-IDENTIFIER: US 5932106 A

TITLE: Process for regeneration of ion-exchange <u>resins</u> used for sugar decolorization, using chloride salts in a sucrose solution alkalinized with calcium hydroxide

#### Abstract Text (1):

Process for regeneration of ion-exchange resins, used in sugar decolorization, using a sucrose solution with sodium or calcium chloride, or another chloride, namely potassium or a mixture of those chlorides, alkalinised with calcium hydroxide. The regeneration of the resin is made with a flow between 1 to 3 cubic meters of a regenerating solution by a cubic meter of resin/hour and a temperature between 40.degree. to 70.degree. centigrade. The sucrose solution used in this resin regeneration process contains between 30 to 600 g of sucrose per liter of solution; calcium hydroxide between 1 to 40 g of CaO per liter of solution; and calcium or sodium chloride, or another chloride, namely the one of potassium, or a mixture of these compounds, in such a quantity that chloride ion concentration is between 3 to 30 g per liter of solution. This regenerating solution is passed through the resin in a quantity between 2 to 4 cubic meters per cubic meter of resin. The effluent from this resin regeneration process, containing sucrose and a low content of chloride ions, can be used, directly or after a chemical or physical treatment, in sugar production or sugar refining processes.

#### Brief Summary Text (2):

The present invention refers to a regeneration process for ion-exchange <u>resins</u> used in the decolorization of sugar solutions in cane or beet sugar factories, sugar refineries, or in industries using sugar.

### Brief Summary Text (4):

In the classic process for regeneration sugar decolorization <u>resins</u>, a solution of 10% of sodium chloride alkalinized with sodium hydroxide is used. However, this process of regeneration is not efficient in colorants removal from <u>resin</u> and produces polluted effluents. In fact, salt effluents resulting from <u>resins</u> regeneration by alkalinized solutions of sodium chloride cause a big problem because they have a high content of organic compounds with a high content of sodium chloride.

#### Brief Summary Text (6):

In the innovative process that is presented here, the <u>resin</u> regeneration is done with a sucrose solution alkalinized with calcium hydroxide, containing a small quantity of calcium chloride or sodium chloride, or another chloride, for example, potassium chloride or a mixture of those chlorides.

## Brief Summary Text (7):

The advantage of this process is that the solution resulting from the regeneration, containing sucrose, can be used in sugar production or sugar refining, without causing pollution problems, as happens in the classic process of <a href="resin">resin</a> regeneration.

# Brief Summary Text (11):

at this low salts concentration resin do not shrink as much as in the classic

sodium chloride regeneration;

#### Brief Summary Text (14):

In view of these facts, and in base of the present process we verify that we can regenerate <u>resins</u>, that is, remove the majority of the colorants fixed to the <u>resin</u> after the decolorization stepby using a sucrose solution with calcium hydroxide and a small quantity of chloride ions.

#### Brief Summary Text (16):

The sucrose solution, resulting from this new regeneration process, containing calcium hydroxide, chloride salts and colorants removed from the <u>resin</u>, can be returned to the sugar production or sugar refining processes, directly, or after treatment with chemicals or through tangential filtration.

#### Brief Summary Text (19):

Ion-exchange  $\underline{\text{resin}}$  to be regenerated using the regeneration process described in this patent must be contained in an appropriate column or columns for a sugar decolorization process.

## Brief Summary Text (20):

During the decolorization step, sugar solutions are fed to the <u>resin</u> column in an up-flow or down-flow way depending on the equipment used.

#### Brief Summary Text (21):

After the decolorization step, sugar solution inside the column is dislocated with water. During this procedure sucrose concentration in the column effluent decreases. When sucrose concentration reaches a value identical to sucrose in the regenerating solution, resin regeneration starts. For resin regeneration, a sucrose solution containing calcium hydroxide and sodium or calcium chloride or other chlorides, is fed to the resin column in a flow between 1 to 3 cubic meters per cubic meter of resin per hour and at a temperature between 40.degree. to 70.degree. C. The quantity of regenerating solution used in this regeneration is between 2 to 4 cubic meters per cubic meter of resin inside the column.

#### Brief Summary Text (22):

After the passage through the  $\underline{\text{resin}}$  of this regenerating solution, the  $\underline{\text{resin}}$  is washed with water, decompressed with air and washed again as usual in the  $\underline{\text{resin}}$  regeneration processes.

## Brief Summary Text (23):

From time to time and as is usual in decolorization <u>resins</u>, an acid regeneration (NaCl+HCl) and alkaline regeneration (NaCl+NaOH) can be performed.

#### Detailed Description Text (2):

This new <u>resin</u> regeneration process was experimentally used to regenerate a styrenic divinyl-benzenic <u>resin</u> with quaternay ammoniom groups, initially in the chloride form. The <u>resin</u>, placed in a one liter column was used to decolorize carbonated liquor from a sugar refinery. After 40 BV (bed volumes) of liquor, the <u>resin</u> was washed and regenerated with this new <u>resin</u> regeneration process. In ten successive liquor cycles and regenerations, <u>resin</u> efficiency was maintained at a high level with decolorization of liquor higher than 90%.

#### Detailed Description Text (3):

In this experiment, the solution effluent from the <u>resin</u> regeneration, with this new process, was treated by <u>nanofiltration</u>. It was observed that more than 90% of colour, measured at 420 nm at pH 7.0, was separated to the retentate. The permeate was used to prepare the <u>regenerating</u> solution, after adding the appropriate quantities of sucrose, calcium hydroxide and calcium chloride. <u>Resin</u> regenerated with this permeate maintained the efficiency above 90% of decolorization.

#### CLAIMS:

- 1. In a process comprising decolorizing a sugar solution by contacting said solution with an ion-exchange <u>resin</u>, and recovering a decolorized sugar solution, wherein the <u>resin</u> is regenerated, the improvement which comprises regenerating said <u>resin</u> by contacting it with a regenerating solution containing 30 to 600 grams of sucrose per liter of solution, calcium hydroxide in a quantity, calculated on the basis of CaO, of between 1 to 40 grams of CaO per liter, and calcium chloride, in a quantity such that the chloride ion concentration is between 3 to 30 grams of chloride per liter solution.
- 2. The process according to claim 1 wherein the <u>resin</u> regeneration is accomplished by flowing the regenerating solution through a body of the <u>resin</u> at a flow rate of between 1 to 3 cubic meters of said regenerating solution per cubic meter of <u>resin</u>, per hour, at a temperature of between 40 and 70.degree. C.
- 3. The process according to claim 2 wherein the amount of regenerating solution employed is between 2 to 4 cubic meters per cubic meter of  $\underline{\text{resin}}$ .
- 4. The process according to claim 1 the amount of regenerating solution employed is between  $2^{\cdot}$  to 4 cubic meters per cubic meter of <u>resin</u>.
- 5. The process according to claim 1 wherein the regenerating solution, after contacting said <u>resin</u>, is subjected to calcium precipitation with carbon dioxide, sodium carbonate, phosphoric acid or sodium phosphate.
- 6. The process according to claim 1 wherein the regenerating solution, after contacting said <u>resin</u>, is subjected to tangential filtration through a filtration medium, resulting in a portion retained in the filtration medium containing the majority of colorants and a portion which permeates the filter medium which is recycled to said process, where it is employed in the production of said regenerating solution.

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